Appendix S. Quality Assurance Audit Team Memorandum
Findings of Initial Laboratory Audit and DPR Response

Air Resources Board

Alan C. Lloyd, Ph.D.
Chairman
2020 L Street • P.O. Box 2815 • Sacramento, California 95812 • www.arb.ca.gov

Winston H. Hickox
Secretary for Environmental Protection

Gray Davis
Governor

MEMORANDUM

TO: Randy Segawa
Environmental Monitoring and Pest Management Branch
Department of Pesticide Regulation

FROM: Michael Miguel, Manager
Quality Assurance Section
Monitoring and Laboratory Division

DATE: October 22, 1999

SUBJECT: PRELIMINARY LABORATORY EVALUATIONS FOR LOMPOC FUMIGANT MONITORING STUDY

On October 12 and 13, 1999, a quality assurance team conducted preliminary on-site evaluations of three laboratories that will analyze air samples of four soil fumigants as part of a pesticide monitoring study in Lompoc. Monitoring is scheduled to start in late October or early November 1999. The quality assurance team was led by Don Fitzell of the Air Resources Board. Other members included Mathew Plate of the U.S. EPA, Kathy Orr of the Department of Pesticide Regulation, Susan Kegley of the Pesticide Action Network, and Lynn Baker of the Air Resources Board.

In order to assist the laboratories in obtaining the best possible data, the team is releasing preliminary findings which it feels should be addressed before monitoring begins. Some of these issues were brought up by the laboratories themselves, some were noted by the audit team. Most if not all of the issues were discussed with the various laboratory personnel, but it was felt that a preliminary summary would help remind the laboratories of these issues. A formal report will be issued at a later date.

- Laboratory and field quality control spikes should be prepared in the same manner by each laboratory involved (e.g., if sorbent tubes are spiked directly into the tubes with the compound in solution, another laboratory should not spike the tubes by passing air over glass wool and then collecting the compound on the sorbent).

- Laboratories analyzing colocated samples should be sure their method is as close as possible to the primary analytical laboratory.

Winston H. Hickox
Secretary for Environmental Protection

Department of Pesticide Regulation

Paul E. Helliker, Director
830 K Street • Sacramento, California 95814-3510 • www.cdpr.ca.gov

Gray Davis
Governor

MEMORANDUM

TO: John S. Sanders, Ph.D.
Chief
Environmental Monitoring and
Pest Management Branch

FROM: Randy Segawa, Senior Environmental Research Scientist
Environmental Monitoring and
Pest Management Branch
(916) 324-4137

DATE: December 1, 1999

SUBJECT: RESPONSE TO LOMPOC PRELIMINARY QUALITY ASSURANCE EVALUATION

The Department of Pesticide Regulation (DPR) has formed the Lompoc Quality Assurance Team to provide an independent evaluation of the field and analytical procedures used for air monitoring in Lompoc. The team includes Don Fitzell, Air Resources Board; Matt Plate, U.S. Environmental Protection Agency; Kathy Orr, DPR; and Susan Kegley, Pesticide Action Network. DPR has arranged for three laboratories to conduct the analysis of fumigant samples, the California Department of Health Services Environmental Health Laboratory (DHS), the California Department of Food and Agriculture Center for Analytical Chemistry (DFA), and the U.S. Environmental Protection Agency Region 9 Laboratory (U.S. EPA). On October 12 and 13, the team conducted a preliminary evaluation of the three laboratories and provided two sets of comments. The following is a response to the comments.

1. If samples are sent FedEx to the Sacramento International Airport, how will the samples be transported from the airport to the DFA lab? Samples should be sent on Monday thru Thursday to avoid being delivered on weekends.

DPR has arranged for the samples to be shipped overnight directly to DFA. DPR has arranged for sampling personnel to ship samples between Monday and Thursday.

2. Will DFA be receiving or needing to prepare trip or field spikes? Blanks and trip spikes seem necessary. What spike level? Should these be blind spikes?

The DFA will receive, but not prepare spikes. DPR has arranged for DHS to prepare trip spikes and ship them to the field. Field personnel will ship the trip spikes to DFA at the same time as field samples. The trip spikes will be blind. In addition, a field blank will accompany each shipment of samples.

3. If DHS confirms samples with a second column, is it necessary for the DFA lab to confirm positive results, since DFA's role is confirmatory?

No

4. The Fumigant Sampling and Analysis Plan (FSAP) should specify frequency for making stock solutions and working standards if there is a concern about consistency between labs.

The FSAP specifies that standards should be prepared at least every six months, consistent with the stability determined by DFA.

5. The FSAP should specify the frequency desired for replicate analysis of samples (e.g., all canisters or a percentage). Each canister run takes about 40 minutes.

At a minimum, U.S. EPA will analyze two canisters from each methyl bromide and 1,3-dichloropropene fumigation monitored. This has been specified in the FSAP (see the sampling schedule provided in Appendix F of the FSAP for details).

6. If DHS is successful in developing an adequately sensitive method for MITC from canisters (charcoal tubes is the primary sampling method), the U.S. EPA lab could attempt to analyze some collocated canisters for MITC as a tentatively identifiable compound. U.S. EPA would need information on where to obtain a MITC standard or would need to borrow some of DHS' standard.

DPR will forward the canister method for methyl isothiocyanate as soon as DHS finalizes the method.

7. DPR should specify in the FSAP the spiking levels for trip and field spikes.

DPR has discussed the spike levels with DHS. However, these samples will be blind so the levels will not be specified in the FSAP.

8. DPR should specify in the FSAP how to report lab data (e.g., uncorrected along with the desorption efficiency data or corrected data). The DFA and DHS labs should be consistent on this.

DPR has arranged for the laboratories to report the unadjusted data as well as the quality control results. This is specified in the FSAP, section 6.5.

9. DHS can confirm MITC samples with GC/MS. What percent of positives does DPR want confirmed? If extracts are too low to detect, should several low extracts be combined in an attempt to be detected by the GC/MS?

DPR has specified in the FSAP that DHS confirm with GC/MS, all samples at or above the acute NOEL for all analytes. The acute NOELs can all be quantified with this method.

10. Samples should be sent Monday thru Thursday to avoid delivery of samples on weekends.

DPR has arranged for the samples to be shipped overnight directly to each laboratory. DPR has arranged for sampling personnel to ship samples between Monday and Thursday. This has been specified in the FSAP.

11. DHS prefers use of custody seals on the ends of adsorbent tubes collected in the field so that it can be determined if caps came off the sampling tubes during shipping.

DPR will use the custody seals provided by DHS.

12. DHS expressed concern about the viability of using MITC field spikes. Should DHS still proceed with preparing and using MITC field spikes?

DPR and DHS have conducted two trial runs using MITC spikes. Results of these trial runs will be available December 1, 1999 and evaluated by DPR and DHS.

13. DHS prefers use of batch chain of custody forms (DHS gave us a copy) rather than individual chain of custody forms for each adsorbent tube.

DPR will use the batch chain of custody forms provided by DHS.

14. Samples should be placed in separate ice chests with dry ice in the field depending on the destination of the ice chest (DFA or DHS) so that tubes don't need to be separated later. This will allow use of the batch chain of custody forms.

Field personnel will use separate ice chests.

15. What percentage of positive results for all four fumigants should be confirmed with a separate method or column?

Confirmation is to be performed on 10% of *all* samples collected, as described in the FSAP. Confirmation is to be performed by a separate laboratory, as described in the FSAP. It is necessary to confirm positive sample results as well as sample results below the detection limits (i.e. confirm the sample was actually none detect). (See question 9 above for confirmation of positive sample results.)

16. DHS prefers that after sampling and prior to shipping, canisters be stored in a building with more temperature control than a rented storage shed. Does the Lompoc Agricultural Commissioner Office have a back room where canisters could be stored prior to shipping? Is the Agricultural Commissioner Office air conditioned?

The lead field person works for a canister manufacturer. DPR has confirmed with this individual that canisters can be properly stored under winter temperatures in Lompoc at the rented storage facility.

17. DHS recommends that canisters be shipped by ground transportation rather than FedEx air freight. Is ground shipment possible from Lompoc? How many days would it take by ground shipment from Lompoc to Berkeley?

DPR has confirmed with chemists at U.S. EPA that canisters can be safely shipped via air. U.S. EPA confirmed this with the canister manufacturer.

18. Following completion of a sampling run, canister caps should be tightened with a wrench, rather than finger tight.

Field personnel will use a wrench to secure the canister caps.

19. Laboratory and field quality control spikes should be prepared in the same manner by each laboratory involved.

Both laboratories will spike the sorbent tubes directly.

20. Laboratories analyzing collocated samples should be sure their method is as close as possible to the primary analytical method.

DFA has made minor, if any, changes to the methods used by DHS.

21. All laboratories should have a formal internal review of the data prior to releasing it.

The laboratory supervisors will review and approve all data prior to transmittal to DPR.

22. A formal criteria for precision of replicate samples should be established and a course of action stipulated if it is not met.

The FSAP contains the criteria for duplicate, spike, and blank samples.

23. DFA and DHS laboratories have compared standards prior to the start of the study. It is recommended that the laboratories also compare the standards at the end of the study to ensure that no shift or degradation has occurred.

The laboratories will compare standards at the end of the study if the confirmation samples show significant differences, as specified in the FSAP.

24. Differences in desorption techniques were noted amongst the laboratories. It is recommended that each laboratory report its own desorption efficiencies.

Each laboratory will report its own desorption efficiencies.

25. Laboratories using canisters should be sure that certification standards for cleanliness are the same or are very close.

Both laboratories (DHS and U.S. EPA) use the same SOP for cleaning and operating canisters (see FSAP for details).

26. If quantities of cis and trans isomers of Telone are reported separately, it should be checked that the same quantity is obtained when the isomers are calculated together.

Both DHS and DFA will report the cis and trans isomers of 1,3-dichloropropene separately to DPR. They will use the same method of quantitation for the isomers.

If you have any questions, please free to call me.

cc: Lisa Ross
Barbara Bates, U.S. EPA
Steve Wall, DHS
Cathy Cooper, CDFA
Dave Vener, Xontech, Inc.
Don Fitzell, ARB
Matt Plate, U.S. EPA
Susan Kegley, Pesticide Action Network
Kathy Orr, WH&S
Lynn Baker, ARB
Michael Miguel, ARB